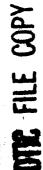


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Victoria, B.C.

Materials Report 81-C

POSITIVE IDENTIFICATION OF MICROGRAM QUANTITIES OF ASBESTOS USING INFRARED SPECTROSCOPY

G.A. Luoma, L.K. Yee and R. Rowland

September 1981



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POSITIVE IDENTIFICATION OF MICROGRAM QUANTITIES OF ASBESTOS USING INFRARED SPECTROSCOPY

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Approved

RESEARCH AND DEVELOPMENT BRANCH DEPARTMENT OF NATIONAL DEFENCE CANADA

ABSTRACT

much recent interest has been expressed in identifying and quantifying small amounts of asbestos in many materials. To date, optical microscopy, x-ray diffraction, scanning electron microscopy coupled with energy-dispersive X-ray spectrometry (SEM-EDS), air particle counting, and Raman spectroscopy have all displayed limited applicability to samples of diverse origin. The present report shows that infrared spectroscopy can be used to identify absolutely microgram quantities of various asbestos types in all normally encountered samples. Its main advantages over other methods include ease of sample preparation, speed, and low cost of necessary equipment. When combined with simple purification procedures, it can be used for largely non-asbestos mixtures. Finally, when combined with present day microcomputer systems, automatic semiquantitative identification of mixtures of different asbestos types can be made.



A

1. INTRODUCTION

In the past ten years, asbestos has been found to cause a number of serious illnesses among mine workers. The major illnesses, asbestosis and mesothethelioma are caused by the direct inhalation of asbestos fibers into the throat and lungs^{1,2}. Therefore, monitoring air samples in areas exposed to asbestos dust is becoming increasingly important. Other experiments suggest that certain types of asbestos belonging to the amphibole class are potentially more dangerous than those belonging to the serpentine class ^{3,4}. This latter observation is directly related to particle size and shape, since amphiboles are usually composed of larger, needlelike fibers which can cause more tissue damage. As a result, determination of asbestos type is also important in assessing potential harmful effects.

As mentioned above, there are two basic classes of asbestos: the serpentines and the amphiboles. The only member of the serpentine class is chrysotile asbestos, and it is obtained almost exclusively from Canada and Rhodesia. It composes approximately 95% of the asbestos found in industrial applications ⁵. The amphibole class contains three commonly encountered species called amosite, crocidolite and anthophyllite, as well as tremolite. The major types and their chemical formulae are contained in Table I.

TABLE I: Commonly occurring types of asbestos

Asbestos Type	Formula	% OH by weight
serpentines chrysotile	Mg ₃ (Si ₂ O ₅)(OH) ₄	25%
amphiboles amosite	$(Mg, Fe^{+2})_7 (Si_8 O_{22}) (OH)_2$	~ 5%
crocidolite	$\text{Na}_{2}\text{Fe}^{+3}_{2}\text{Fe}^{+2}_{3}(\text{Si}_{8}\text{O}_{22})(\text{OH})_{2}$	~ 5%
anthophyllite	$(Mg, Fe^{+2})_7 (Si_8 O_{22}) (OH, F)_2$	~ 2%

Although many asbestos substitutes are currently available, the complete replacement of asbestos has not been achieved ⁶. At present, asbestos is still used in such products as fire blankets, lagging materials for heat pipes and electronics, gaskets and cement. About 3.5 million tons of asbestos are consumed each year, so methods for its identification are still required.

Much current experimentation concerned with developing positive detection of small amounts of asbestos has been carried out. The major methods used are polarized light microscopy, x-ray diffraction and scanning electron microscopy coupled with energy-dispersive X-ray spectrometry (SEM-EDS) 7-10. Other less common methods include thermal methods such as differential scanning calorimetry (DSC) and Raman spectroscopy 11,12. All of these techniques produce very good qualitative, and sometimes quantitative, results for specific analyses, but none has been shown to be generally applicable to all

asbestos analyses. Furthermore, some are time consuming and not truly confirmatory (e.g. light microscopy), others require fairly large samples volumes (e.g. X-ray diffraction), and still others require expensive specialized equipment (e.g. SEM-EDS). Therefore, a fast identification technique having a wide general applicability and requiring only small samples is required.

A few infrared studies of asbestos have been reported ¹³⁻¹⁶.

Unfortunately, most of them have had limited success either because of the low sensitivity of older infrared spectrophotometers or the limited spectral range (4000 to 600 cm⁻¹). Patterson and O'Connor ¹³ originally showed that amosite and crocidolite contain strong and characteristic SiO infrared absorption bands at 1200-900 cm⁻¹, and these bands were also found for chrysotile by Beckett et al¹⁴. For chrysotile asbestos, a further characteristic sharp OH peak at 3670 cm⁻¹ was also reported ^{14,15}. Finally, the region between 800 cm⁻¹ and 200 cm⁻¹ was shown to contain a characteristic pattern of peaks which could be successfully used to classify asbestos types ¹⁶.

The use of infrared spectroscopy for the analysis of mixtures containing asbestos has been hampered by the presence of interfering bands of the non-asbestos matrix¹⁵. However, the advent of computer methods and multiple scanning techniques have resulted in the ability to detect low limits of asbestos and the presence of a small proportion of asbestos in a non-asbestos matrix¹⁶.

In this report, we present infrared data for the common types of asbestos used in industry and by the armed forces. We also show that infrared sensitivity and selectivity are as good as all other techniques, and that infrared spectroscopy can have superior capability when used on mixtures.

II. EXPERIMENTAL:

When preparing asbestos-containing samples for infrared analysis, the amount of pretreatment necessary is dependent on the source and purity of the sample. In all, three different procedures were performed and are briefly described below.

- 1. If the sample of asbestos is relatively pure, or a single fiber can be easily isolated, approximately 100 µg of sample is ground up in a mortar with approximately 10 mg of potassium bromide (KBr). The resulting mixture is pressed into a 5 mm diameter pellet and inserted directly into the infrared spectrophotometer. If small sample sizes are necessary, as little as a few micrograms of asbestos can be analysed in a 3 mm diameter pellet using a beam condenser attachment.
- 2. If the sample contains large amounts of impurities which produce interfering infrared bands, the high resistance of asbestos to acids and bases can be used to purify it from contaminating organic and metal components. The following method was successfully employed.
 - (a) Stir the asbestos sample (preground) into twenty volumes of concentrated sulfuric acid and suction filter on a sintered glass funnel. Wash with distilled water.
 - (b) Stir the partly purified asbestos in twenty volumes of concentrated ammonia. Wash with distilled water.
 - (c) Rinse the pure asbestos with twenty volumes of acetone and filter. Oven dry at 120°C.

The sample can then be ground and pelletized as before. If the weights of the original and final purified samples are recorded, a semi-quantitative estimate of the proportion of asbestos in the original sample can be made.

3. In some cases, impurities in the asbestos-containing samples are best removed by pyrolysis. In such cases, a small amount of sample is placed in a crucible and is heated on a bunsen flame until all impurities are driven off. However, this procedure is less desirable since pyrolysis alters the asbestos, and produces an inferior infrared spectrum (see Section IIIF).

Pure asbestos samples were obtained from Duke Scientific. All infrared spectra were recorded on a Perkin-Elmer 398 Infrared Spectrophotometer equipped with a Model 3600 Data Station.

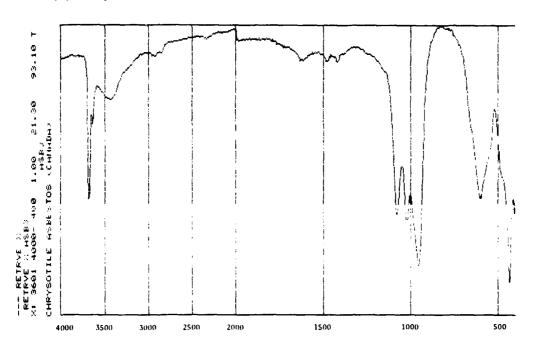
III. RESULTS:

A. Infrared Spectra of Serpentine and Amphibole Standards and Their Characteristic Bands.

As mentioned in the introduction, there are two classes of asbestos; the serpentine class made up of chrysotile, and the amphibole class containing amosite, crocidolite and anthophyllite. The infrared spectra of each type are shown in Figure 1.

Figure 1. Some commonly used asbestos types.

(a) chrysotile asbestos from Canada



(b) chrysotile asbestos from Rhodesia

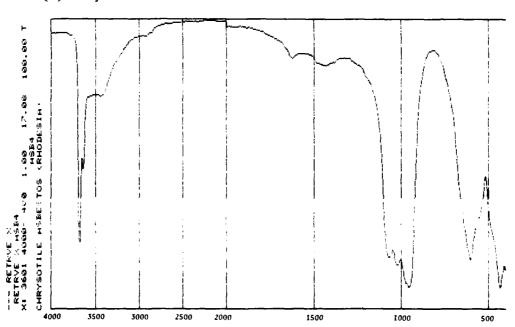
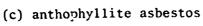
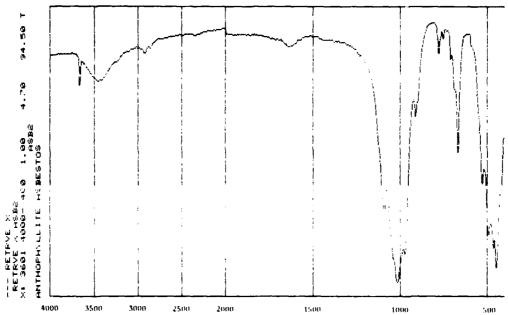


figure 1 (cont.)...





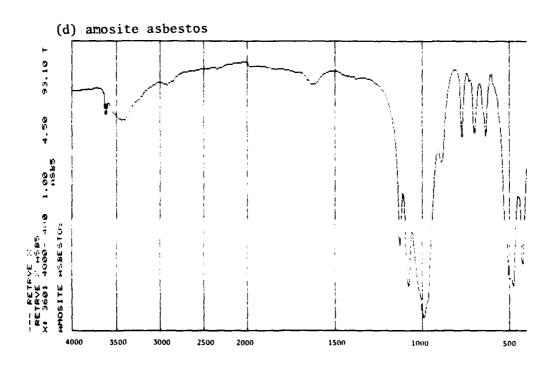
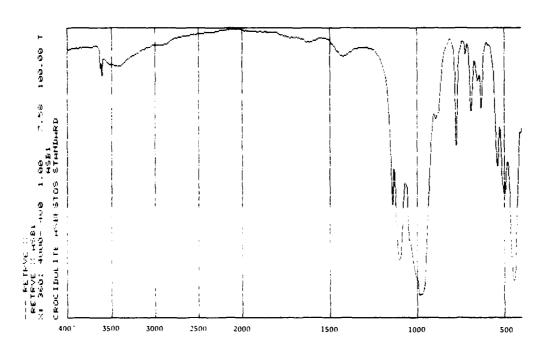


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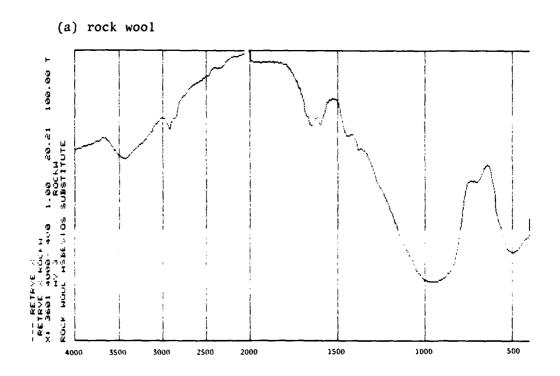
(e) crocidolite asbestos



B. Infrared Spectra of Some Common Asbestos Substitutes.

Asbestos substitutes are generally of three types: glass fiber cloths, man-made fiber cloths impregnated with fire-retardant chemicals, and natural non-asbestos mineral blends. Some infrared spectra of representative samples of each type of substitute are contained in Figure 2.

Figure 2. The infrared spectra of some commonly used asbestos substitutes.



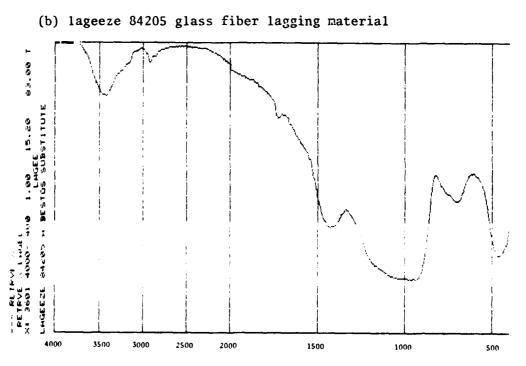
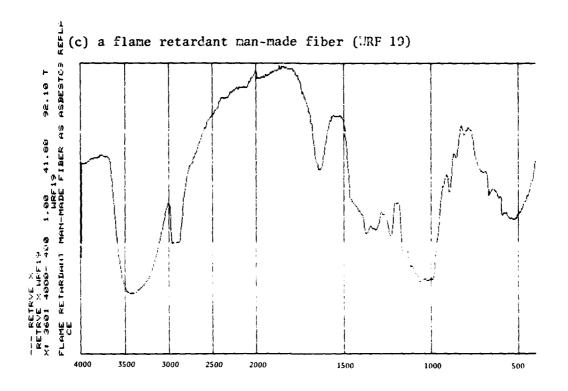


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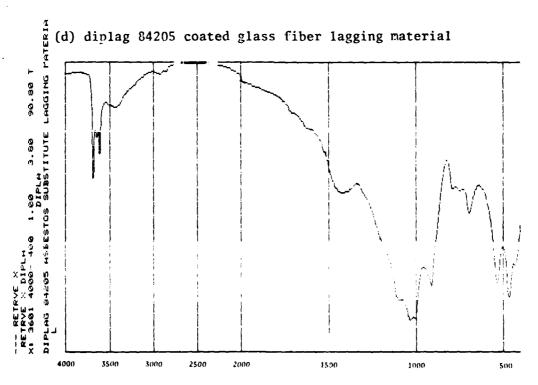
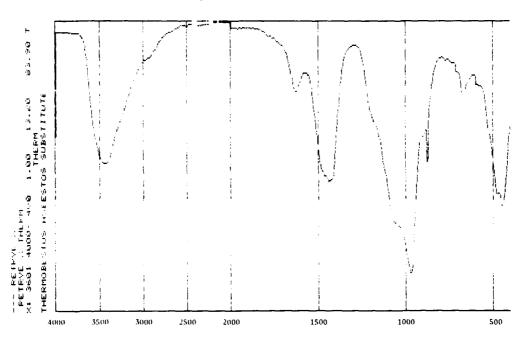


figure 2 (cont.)...

(e) thermobestos inorganic substitute



(f) atlacite inorganic substitute

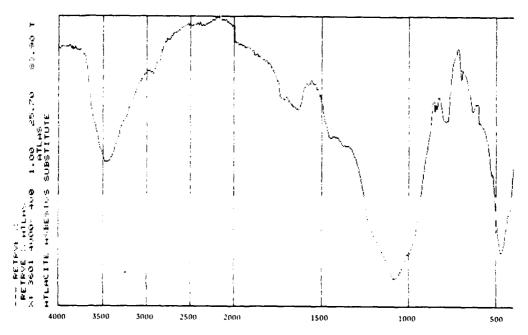
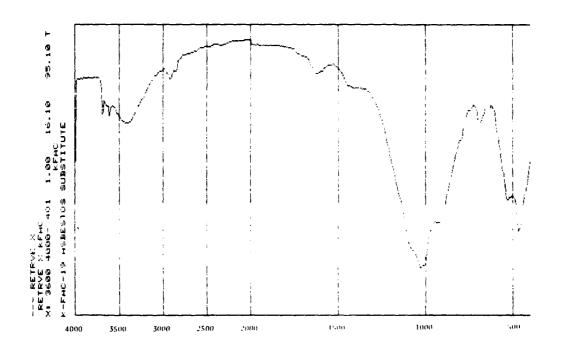


figure 2 (cont.)...

(g) K-FAC-19



Analyses of these spectra show that most amphiboles have a characteristic band between 770cm⁻¹ and 780 cm⁻¹, which is absent in chrysotile asbestos. Furthermore chrysotile asbestos has a characteristic strongly absorbing doublet around 3600-3700 cm⁻¹, which is reduced in intensity or absent in the amphiboles. This doublet is due to OH vibrations of bound hydroxyls in the crystal structure, and the chrysotile asbestos has a higher proportion of OH groups than the amphiboles, resulting in the increased intensity. Finally, chrysotile asbestos lacks any sharp absorption bands between 900 cm⁻¹ and 650 cm⁻¹, while the amphiboles each have characteristic peaks in this region. Therefore, the differences between chrysotile and amphibole asbestos types are readily discriminated.

Among the amphiboles, crocidolite and amosite have a sharp peak between 1120 cm⁻¹ and 1140 cm⁻¹, while such a band is conspicuously absent in the anthophyllite asbestos spectrum. Furthermore, the three amphiboles have unique patterns of sharp bands between 800 cm⁻¹ and 400 cm⁻¹ which make identification simple. The characteristic pattern of these bands is due to the variety of SiO stretching and bending vibrations for various crystal structures.

Although chrysotile is the only serpentine asbestos found, its infrared spectrum varies with place of origin. Thus, Canadian chrysotile is characterized by sharper bands, and the more intense peak at 430 cm⁻¹, in contrast to the chrysotile from Rhodesia. The positions of major bands for each of the asbestos types is contained in Table. 2.

TABLE II: Infrared Peaks of major asbestos types

Asbestos Type						
Crocidolite	Amosite	Anthophyllite		Chrysotile II		
3627 cm ⁻¹	3629 cm ⁻¹	3667 cm ⁻¹	3681 cm ⁻¹	3681 cm ⁻¹		
3611	3609	3547	3637	3637		
3416	3511	3413	3434	3434		
1141	3410	2849	3380	3380		
1100	3236	1257	1079	1079		
986	1617	1094	1022	1022		
973	1126	1015	954	954		
894	1080	975	721	721		
777	993	912	605	605		
725	888	781	600	600		
692	773	755	435	435		
655	729	712				
634	701	670				
541	637	531				
501	495	495				
447	480	465				
	425	450	<u></u>			

* Chrysotile I: Chrysotile asbestos from Canada

Chrysotile II: Chrysotile from Rhodesia

The infrared spectra of the glass fiber substitutes are characterized by very broad unresolved bands. The most characteristic bands are the broad OH stretch at 3400 cm^{-1} , the SiO band at $1200-900 \text{ cm}^{-1}$ and the band at 500 cm^{-1} .

The infrared spectra of the man-made fiber substitutes are characterized by contributions from both the fiber matrix and the fire retardant chemical. The most noticeable differences between these and the other substitutes are the presence of the CH stretch bands around 2900 cm⁻¹ and the large number of sharper peaks which are characteristic of organic components.

The infrared spectra of non-asbestos mineral substitutes contain bands between 1200 cm⁻¹ and 800 cm⁻¹ characteristic of silicates. They also contain broad OH bands at 3400 cm⁻¹. Finally, some, (e.g. thermobestos), contain a strong broad band at 1450 cm⁻¹ and a sharp band at 870 cm⁻¹ which are characteristic of the presence of a carbonate component.

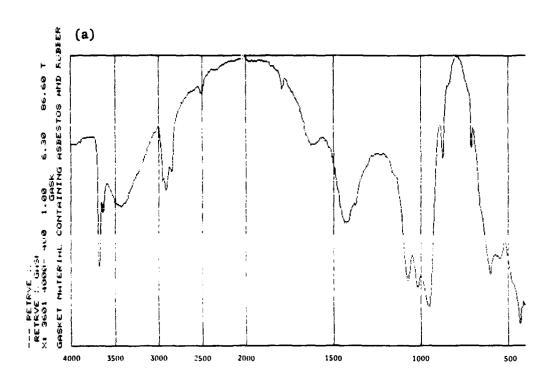
In summary, all of the asbestos substitutes produce characteristic information from their infrared spectra. The spectra are substantially different from those of any of the asbestos types, and they can be readily differentiated as non-asbestos materials.

C. Infrared Analyses of Samples Containing Asbestos and Non-Asbestos Components.

To test the utility of infrared spectroscopy for the analysis of samples largely composed of non-asbestos materials three typical products were analyzed: a) an asbestos-containing rubber gasket used in steam drums of naval vessels; b) a mixture of diatomaceous earth containing 10% asbestos; and c) an asbestos fire blanket containing a chemical binder. In all cases, spectra were recorded both before and after acid/base washing and

are shown in Figures 3-5.

Figure 3. (a) A spectrum of a boiler gasket material composed of asbestosimpregnated rubber. (b) The same gasket material after washing
with sulfuric acid, ammonia and acetone to remove organic components. Note how the washing procedure produces a high quality
asbestos spectrum.



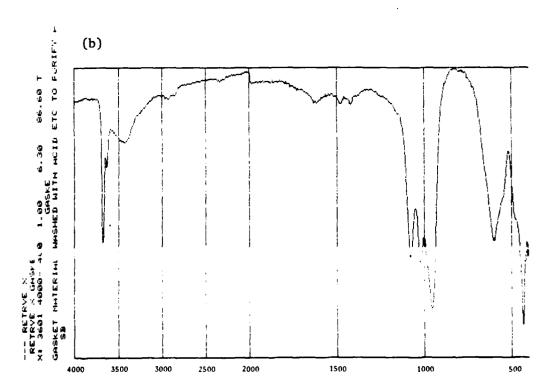
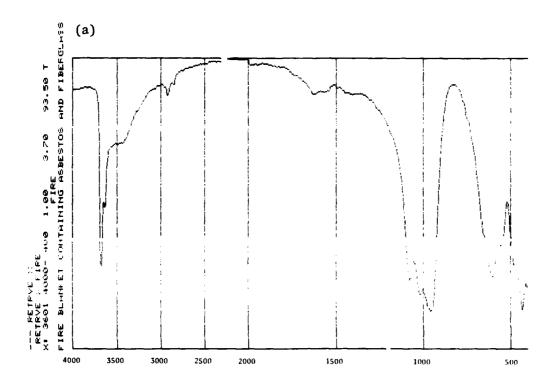


Figure 4. (a) An infrared spectrum of fire blanket material containing fiberglass and asbestos and a small amount of organic binder.

(b) The same blanket material after washing.



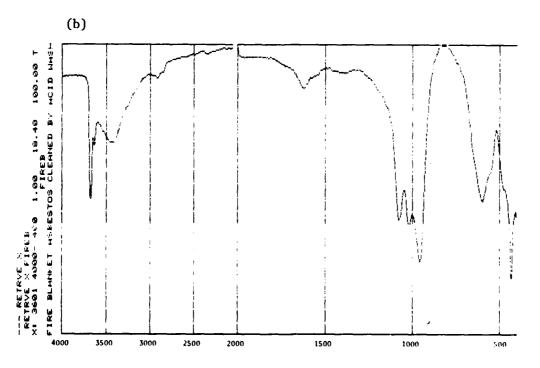
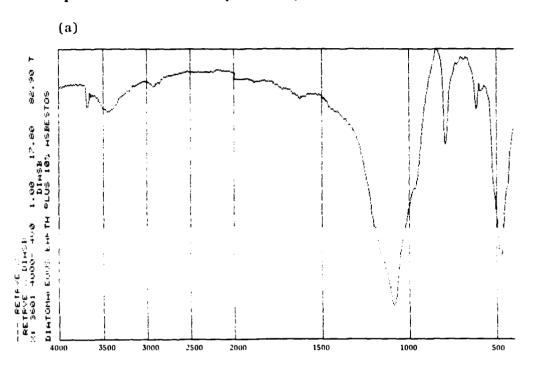
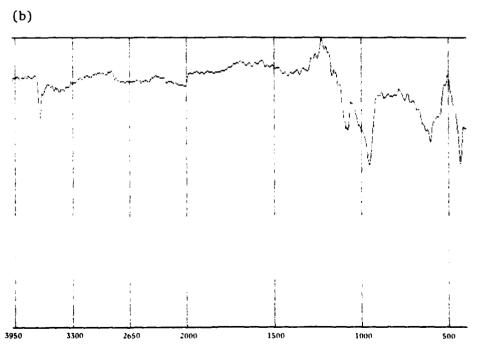


Figure 5. (a) The infrared spectrum of a sample of diatomaceous earth containing 10% asbestos. Note that the asbestos spectrum is almost completely masked by the diatomaceous earth spectrum.

(b) The same spectrum as in (a) except the spectrum of diatomaceous earth has been computer subtracted to produce a difference spectrum which can be positively identified as asbestos.





shows definitively the presence of chrysotile asbestos (cf. Figure 1(a)), as well as an organic component (bands at 2900 cm⁻¹) which is known to be natural rubber. The characteristic asbestos bands show very little interference from the infrared bands of the rubber component. Furthermore, purification by acid/base washing causes the labber component to be removed from the asbestos, without affecting the structure of the asbestos. The resulting infrared spectrum is identical to that of pure Canadian chrysotile asbestos.

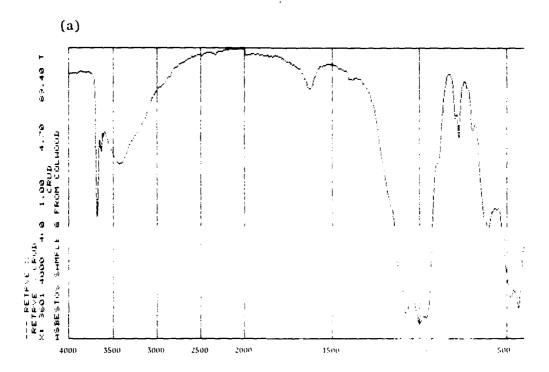
The spectra of the fire blanket before and after treatment are contained in Figure 4. Again the presence of chrysetile asbestos is confirmed even in the untreated blanket. Also, two isolated bands at 2900 cm⁻¹ and 790 cm⁻¹ indicate the presence of the organic binding agent. The acid/base washing eliminated this organic compound, and the resulting infrared spectrum is identical to chrysotile asbestos.

Finally, to show the power of infrared spectroscopy when combined with modern computer accessories, a synthetic mixture of 10% asbestos in diatomaceous earth (amorphous silica) was analysed (Figure 5). The spectra of the mixture and pure diatomaceous earth are very similar. This is because of the similarity of the chemical compositions of amorphous silica and asbestos, and the large infrared contribution of the silica masks the asbestos spectrum. Furthermore, the acid and base washing procedure would not remove the silica from the sample. However, when the difference spectrum of the mixture minus the diatomaceous earth is obtained using computer subtraction methods (Figure 5(b)), the resultant is a spectrum of the asbestos portion of the mixture. Although the resolution and signal-to-noise of the difference spectrum is reduced, it can still be positively identified as chrysotile asbestos.

D. Infrared Spectra of Mixtures of Asbestos Types

As well as identifying asbestos as a minor component in a non-asbestos matrix, mixtures of two or more asbestos types can be analysed by infrared spectra using computer-aided spectral additions (Figure 6). In this case, a suspected asbestos mixture submitted for analysis by Canadian Forces personnel was used.

Figure 6. (a) The infrared spectrum of a sample containing two kinds of asbestos and a third non-asbestos component. (b) A computer simulated spectrum was composed of 40% chrysotile asbestos, 30% amosite asbestos and 30% diatomaceous earth. Note how this simulation not only allows the major components to be identified but also to be semi-quantitatively determined.



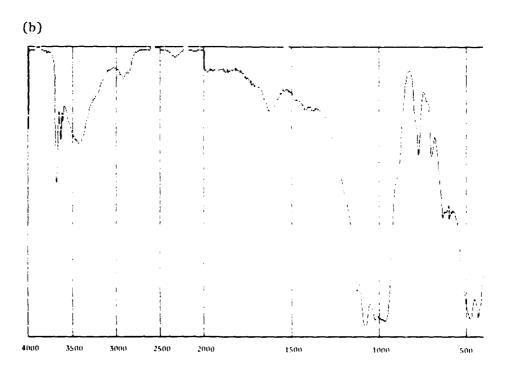


Figure 6(a) is the experimental spectrum, and it contains the typical OH vibrations at 3620 and 3670 cm⁻¹ due to the presence of chrysotile asbestos. However, the SiO region of the spectrum (1200 cm⁻¹ to 900 cm⁻¹) is not typical of chrysotile asbestos alone. Furthermore, the region of 800-600 cm⁻¹ contains some sharp bands typical of amphibole type asbestos, especially the band at 775 cm⁻¹. Figure 6(h) shows a computer simulated spectrum obtained by combining spectra of chrysotile and amosite asbestoses.

As can be seen, the experimental spectrum is well matched by a simulated spectrum consisting of approximately 40% chrysotile asbestos, 30% amosite asbestos, and 30% diatomaceous earth. The presence of both amosite and chrysotile asbestoses was confirmed by x-ray diffraction, but the diatomaceous earth cannot be detected because of its amorphous character. Therefore, for this example, infrared analysis proved superior for estimating quantities of components in a mixture containing multiple types of assestos or non-asbestos materials.

E. The Detection Limit of Asbestos by Infrared Spectroscopy
Since allowed levels of contamination by asbestos in
working environments are low, a useful analytic technique must be able to
accurately detect small quantities of asbestos. Therefore, the detection
limit by infrared spectroscopy was investigated. Figure 7 contains the
spectrum of 10 µg of chrysotile asbestos pelletized in 3 mg of potassium
bromide using a Perkin Elmer beam condenser accessory and a 3 mm pellet. The
figure shows that even a single scan of this small amount produces a high
quality spectrum. The amount of sample used can be further reduced by
using a 1 mm diameter micropellet and a spectrum with substantially less
signal-to-noise could be tolerated. Therefore, infrared spectroscopy can

positively identify asbestos in sub-microgram quantities.

Figure 7. The sensitivity of infrared for detection of microgram quantities of asbestos is demonstrated. The figure shows that a single scan of a sample of 10 micrograms of asbestos produces a high quality spectrum using a beam condenser accessory.

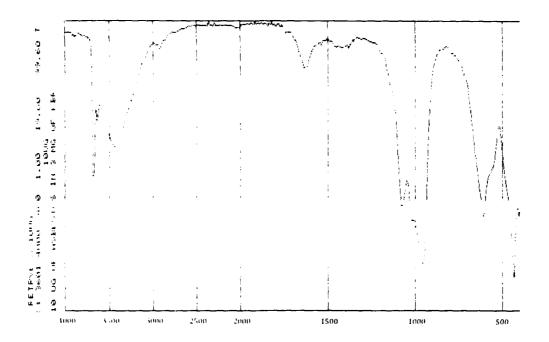
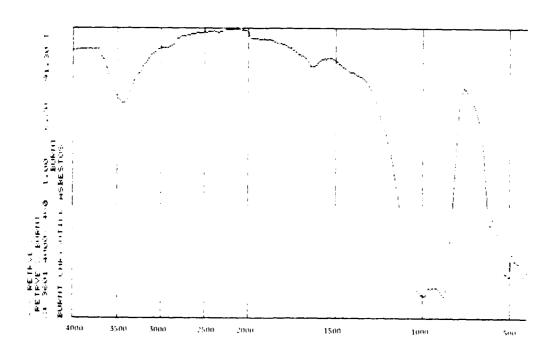


Figure 8. The infrared spectrum of burnt chrysotile asbestos. Note the loss of both resolution and the characteristic OH bands.



F. Pyrolysis of Asbestos and Infrared Analysis

In some samples containing asbestos, pyrolysis may be a more convenient way to purify the asbestos sample. However, x-ray diffraction studies suggested that the asbestos structure is altered by pyrolysis. The infrared spectra of pyrolysed chrysotile asbestos confirms that a structural change has taken place, resulting in the loss of the characteristic sharp peaks at 3620 cm⁻¹ and 3670 cm⁻¹ (Figure 8). Also the other peaks are shifted and less well resolved. Therefore, pyrolysis should be used with infrared analysis of asbestos only when necessary to remove interferences by other components.

IV. DISCUSSION AND CONCLUSIONS:

The above results indicate the utility of infrared spectroscopy for the identification of asbestos materials. Not only can mixtures of asbestos in non-asbestos matrices be analysed, but mixtures of various types of asbestos can be compared to simulated spectra. The detection limit is extremely low (less than 1 µg) for a single infrared scan in a micropellet. This low detection limit can be further increased by the use of multiple scan accumulations in modern dispersive instruments, and by the use of Fourier Transform Infrared Spectrometers.

The main advantages of infrared analysis are three fold:

- It gives positive identification of every kind of asbestos in almost all samples. The only exceptions are extremely small proportions of asbestos in similar compounds which cannot be removed by acid and base pretreatments.
- 2. Infrared analyses as asbestos samples are fast. In most cases positive identification can be obtained in ten minutes.
- 3. The use of minicomputers can automate the identification procedure. Infrared spectra of asbestos standards can be stored on computer discs, and experimental spectra can be automatically compared and identified with these files.

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13. ABSTRACT	•					

Because it is highly toxic and carcinogenic to exposed workers, much recent interest has been expressed in identifying and quantifying small amounts of asbestos in many materials. The present report shows that infrared spectroscopy can be used to identify absolutely microgram quantities of various asbestos types in all normally encountered samples. When combined with simple purification procedures, it can be used for largely non-asbestos mixtures. Finally, when combined with present day microcomputer systems, automatic semiquantitative identification of mixtures of different asbestos types can be made.

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